

## Thymol-blue dyed poly(vinyl butyral) films for monitoring ultraviolet radiation

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### Abstract

Dyed poly(vinyl butyral) (PVB) films prepared by a simple technique of casting alcoholic solution of PVB incorporating thymol blue (TB) indicator and chloral hydrate [ $\text{CCl}_3\text{CH}(\text{OH})_2$ , 2,2,2-trichloroethane-1,1-diol] on a horizontal glass plate are useful for ultraviolet radiation monitoring. These plastic films undergo colour change from yellow to red upon exposure to UV irradiation. The radiation-induced change in colour was analysed spectrophotometrically at the maximum of the visible absorption band peaking at 551 nm. These films have a pronounced response to the main UV radiation spectral regions [UVA (400–320 nm), UVB (320–280 nm) and UVC (280–180 nm)] showing a maximum sensitivity at 200 nm. The measurement uncertainty of estimating ultraviolet radiation energy incident per unit area on the films is found to be 6.6% at a confidence limit of 95% ( $2\sigma$ ). The study of the effect of radiance exposure, irradiation wavelength, chloral hydrate concentration and the post-irradiation stability in different storage conditions has been carried out to characterise the use of these films for actinometric monitoring ultraviolet radiation. © 2000 Elsevier Science B.V. All rights reserved.

**Keywords:** Thymol blue; Chloral hydrate; Poly(vinyl butyral); UV-sensitive indicator

### 1. Introduction

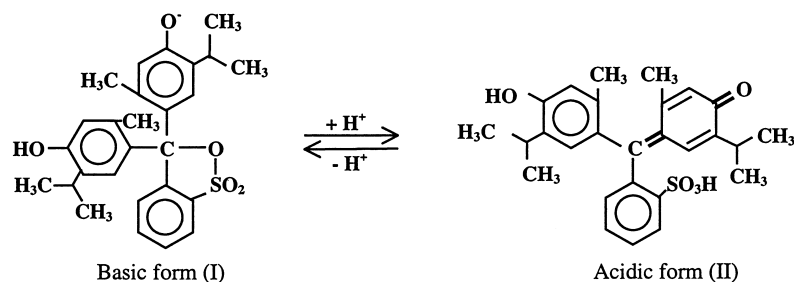
UV radiation has adverse (e.g. skin ageing), as well as beneficial effects (e.g. Vitamin D production), on human health, specifically for workers professionally exposed to UV radiation and generally for the total population. The biological effects of UV radiation in humans are limited to the skin and the eye because of its low penetrating properties in human tissues [1–4]. The normal responses of the skin to UV radiation may be classified as acute, e.g. erythema, melanin pigmentation and Vitamin D production, or chronic, e.g. skin ageing and skin cancer [5]. Erythema (the reddening of the skin sunburn) is a photochemical response of the skin normally resulting from overexposure to wavelengths in the UVC and UVB regions (180–320 nm). Erythema induced by the longer UVB wavelengths (280–320 nm) is more severe and persists for a longer period than that induced by shorter wavelengths [6]. The UVC region has a high penetrating power, and so causes most of the significant adverse health effects, such as skin ageing, skin cancer and eye photokeratities [3]. The UVA (400–320 nm)

is less harmful on humans, however, most chemical effects in the lens protein, tryptophan, have been shown to occur after UVA exposure of the human lens. These effects lead to the formation of chromatic photoproducts that bind to, and alter, the solubility of lens proteins, resulting in a yellowing of the lens materials [7]. Therefore, the measurement of UV irradiance is not only a periodic necessity, but also ensures that work with UV radiation can be carried out safely.

Many substances in the form of dyed and undyed plastic films, exhibiting a measurable change in their properties upon exposure to UV radiation have been investigated in the search for a UV-badge dosimeter, e.g. polysulphone film [8,9], diazo films [10,11], and polyvinyl chloride (PVC) films incorporating photosensitizing drugs [12–14]. Recently, radiochromic films have been studied for use in UV-radiation actinometry [15–18]. UV-sensitive indicators based on poly(vinyl butyral) incorporating an acid sensitive dye (bromophenol blue) and an acid releasing agent (chloral hydrate) have been developed [19].

It is known that acid–base indicators can exist in equilibrium between two tautomeric forms having different colours and the ratio of the two forms depends on the concentration of hydrogen ion in the medium. In our case, thymol blue (TB) is yellow at  $2.8 < \text{pH} < 8$  (form I) and

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Scheme 1.

red at  $\text{pH} < 1.2$  (form II) as represented by the schematic diagram (Scheme 1).

In the present work, flexible free-standing polymeric thin films have been prepared by casting PVB solutions containing thymol blue and chloral hydrate; these were evaluated as UV-sensitive indicators. The assessment of uncertainties, effect of both irradiation wavelength and chloral hydrate concentration on the performance of TB/PVB films as well as post-irradiation stability at different storage conditions have been investigated.

## 2. Experimental procedures

### 2.1. Preparation of stock solution of indicator

The stock solution of the sodium salt of the indicator was prepared by dissolving 0.08 g of thymol blue indicator (TB) (product of CHEMAPOL, Czech Republic) in 1.7 ml of an aqueous solution of NaOH ( $[\text{NaOH}] = 0.1 \text{ mol/l}$ ) and then the volume was completed by ethanol in a 50 ml volumetric flask. It should be noted that the TB stock solution is almost neutral (the pH was found to be 6.8) where the NaOH used in its preparation is an equivalent concentration to prepare the sodium salt of TB.

### 2.2. Preparation of the films

Poly(vinyl butyral) has previously been used successfully to make free-standing dyed films cast on a flat glass surface, from which they can be stripped as flexible foils [19–21]. The 5 g of poly(vinyl butyral) (PVB) (Pioloform BM18, product of Wacker Co., USA) were well dissolved in 100 ml of *n*-butanol at about  $50^\circ\text{C}$ . The solution was kept well stirred at the same temperature for about 24 h, left to cool and then divided into four parts each one of 25 ml volume. The 3 ml of TB stock solution was added to each 25 ml part. The 0.5, 1.0, or 3.0 g of chloral hydrate (product of Merck, Germany) were added to three parts and the fourth one was left without chloral hydrate. The four solutions were kept well stirred at room temperature for about 3 h in order to obtain a uniformly mixed solution. Each solution was poured onto a  $15 \text{ cm} \times 15 \text{ cm}$  horizontal glass plate and dried at room temperature for about 48 h. Four films were obtained

with the same concentration of TB ( $0.33 \text{ phr}^1$ ) and different concentrations of chloral hydrate (0, 34, 68 and  $205 \text{ phr}$ ). After drying, the films were stripped from the glass plate, then cut into  $1 \text{ cm} \times 1 \text{ cm}$  pieces and stored for different investigations. The thickness of the obtained films was found to be  $0.075 \pm 0.005 \text{ mm}$  ( $1\sigma$ ).

### 2.3. Irradiation procedure and apparatus

For calibration purposes and irradiation of samples, a standard 180 W mercury lamp (type EMITA VP-60; made in Poland) was used. Monochromatic filters (bandwidth, 5 nm) at 200, 248.5, 298.8 and 366 nm (Oriel Corporation, Stratford, CT, USA) were used to provide the required irradiation wavelength. Intensity meters for short- and long-wave UV (models J-225 and J-221, Ultraviolet Products, Inc., San Gabriel, CA, USA) were used to measure the intensity of UV light from the mercury lamp. The sample was covered by a filter and then fixed at a certain distance within the path of the UV lamp radiation.

A Unicam UV4 spectrophotometer (product of Unicam Co. Ltd., UK) was used to measure the absorption spectra of the unirradiated and irradiated films.

## 3. Results and discussion

### 3.1. Radiation-induced absorption spectra

The absorption spectra of TB/PVB films (containing  $34 \text{ phr}$  chloral hydrate), unirradiated or irradiated to different doses at an irradiation wavelength of  $298.8 \pm 5 \text{ nm}$  are shown in Fig. 1. Similar absorption spectra at irradiation wavelengths  $201.2 \pm 5$ ,  $248.5 \pm 5$  and  $366 \pm 5 \text{ nm}$  were obtained. Also, absorption spectra of TB/PVB films containing 68 and  $205 \text{ phr}$  chloral hydrate at the same irradiation wavelengths were obtained.

On UV irradiation the yellow TB/PVB film shows a significant colour change to red, indicating the transformation of TB to its acidic form. The absorption spectrum of the unirradiated yellow TB/PVB film (curve 1, Fig. 1) shows a

<sup>1</sup> Here  $\text{phr}$  = part per hundred parts by weight of resin.

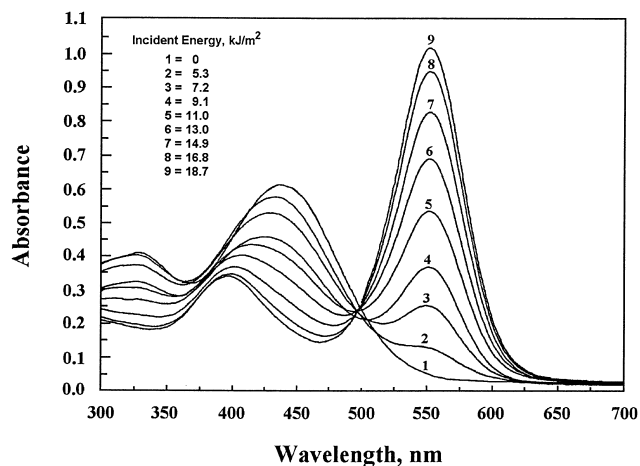


Fig. 1. Variation in the absorption spectrum of TB/PVB films ([chloral hydrate] = 34 phr) with incident UV dose at an irradiation wavelength of  $298.8 \pm 5$  nm.

maximum absorbance at 435 nm. The amplitude of this absorption band at 435 nm (characteristic of the yellow colour) decreases gradually and shifts to lower wavelengths with the increase of the UV-radiation dose, at the same time a red colour (represented by an absorption band at 551 nm) begins to develop and the intensity of this colour increases with the increase of incident UV-radiation. It can be seen that, the radiation-induced change in colour from yellow to red takes place through an isobestic point at about 500 nm wavelength.

It has been found experimentally that films of PVB alone and films of TB/PVB without chloral hydrate do not undergo a change in colour or absorbance on UV irradiation within the dose range studied. Therefore, it can be concluded that chloral hydrate is responsible for the change in colour of TB/PVB films. In other words, UV irradiation of TB/PVB films containing chloral hydrate probably produces HCl, which changes the indicator to its acidic form, leading to a change in colour of the TB indicator.

An examination of the change in absorbance  $\Delta A$  ( $\Delta A = |A_i - A_o|$ , where  $A_o$  and  $A_i$  are the absorbances before and after irradiation, respectively) as a function of UV incident energy indicates that the largest changes occur at 551 nm. Therefore, this spectrophotometric wavelength was subsequently used to quantify UV-induced changes in TB/PVB film at different irradiation wavelengths.

### 3.2. Dose and wavelength responses

The UV irradiations were carried out at  $201.2 \pm 5$ ,  $248.5 \pm 5$ ,  $298.8 \pm 5$  and  $366 \pm 5$  nm irradiation wavelengths. It was found experimentally that UV radiation penetrates the film and the intensity of the penetrated radiation increases with the increase of both irradiation wavelength and irradiation time. For example, at the beginning of irradiation with the irradiation wavelength 201 nm (the lowest penetrating wavelength in this film), the intensity of the penetrated radiation through one film was

about 5% from its initial value and increases gradually to reach about 60% after irradiation for  $1 \text{ kJ m}^{-2}$ . This may be due to the absorption of the energy of the irradiation wavelength by the film for the chemical reaction but the penetration increases gradually with the increase of irradiation time as the reaction goes to end. Also, the dependence of the penetration on the irradiation wavelength reflects the sensitivity of the radiation-induced chemical reaction in the film towards the wavelength of incident radiation. It can be concluded that the sensitivity of the films to UV radiation decreases with the increase of irradiation wavelength.

In order to establish the dose response curves of TB/PVB films containing different concentrations of chloral hydrate (34, 68, 205 phr) to UV radiation, the change in absorbance ( $\Delta A$ ) at 551 nm wavelength was investigated as a function of incident UV dose (four films at each dose) for different irradiation wavelengths ( $201 \pm 5$ ,  $248.5 \pm 5$ ,  $298.8 \pm 5$  and  $366 \pm 5$  nm). Fig. 2 shows the results obtained for TB/PVB films containing 34 phr chloral hydrate, at 201, 248.5, 298.8 and 366 nm irradiation wavelengths. Similar response curves for TB/PVB films containing 68, 205 phr chloral hydrate at the same irradiation wavelengths were obtained. The results show that the sensitivity of the films to UV radiation decreases with increasing irradiation wavelength. In addition, the response at all four irradiation wavelengths is non-linear (S-shape) and tends to saturate at high UV exposure doses. It may be observed from the data illustrated in Fig. 2 that the initial part of the curve depends on the UV irradiation wavelength. The flattening of this portion of the curve decreases with decreasing irradiation wavelength. Accordingly, this behaviour may be attributed to the  $[\text{H}^+]$  formed on irradiation, as its concentration is expected to increase with

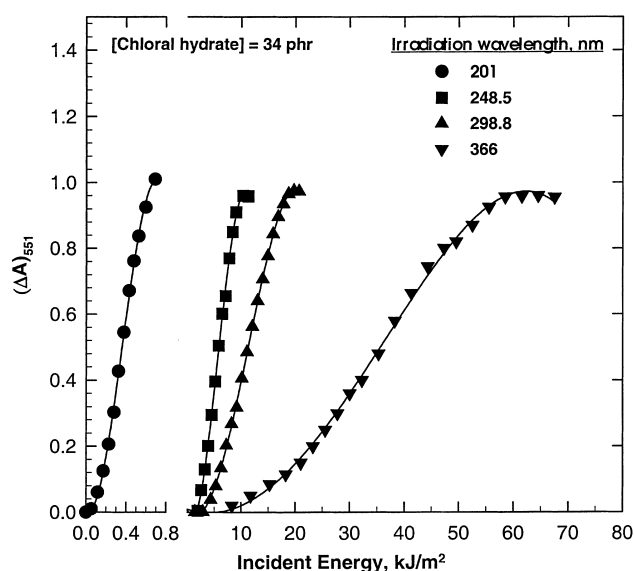


Fig. 2. Variation in  $(\Delta A)_{551}$  at 551 nm of TB/PVB films ([chloral hydrate] = 34 phr) with incident UV dose at different irradiation wavelengths.

Table 1

The constants  $a$ ,  $b$ ,  $c$  and  $d$  for TB/PVB films ([chloral hydrate] = 34 phr)

$\lambda$ (nm)	$a$	$b$	$c$	$d$
201.2	0.013	1.16	−1.27	0.81
248.5	0.955	22.06	−39.74	29.13
298.8	1.915	36.6	−53.7	35.68
366	4.53	136.2	−226.89	154.92

decreasing UV irradiation wavelength. Therefore, a certain  $[H^+]$  level is required, corresponding to a certain pH value, in order for the indicator TB to start to change colour. The final non-linear section of the curve may be ascribed to the complete transformation of the indicator from the basic to the acidic form.

The curves in Fig. 2 for the TB/PVB film containing 34 phr chloral hydrate irradiated at 201.2, 248.5, 298.8 and 366 nm were fitted with third-order polynomial functions giving the following general relationship:

$$D = a + bx - cx^2 + dx^3 \quad (1)$$

where  $D$  is the UV incident energy ( $\text{kJ m}^{-2}$ ),  $x = \Delta A_{551}(D, \lambda)$  (the change in absorbance measured at 551 nm for a UV exposure energy  $D$  ( $\text{kJ m}^{-2}$ ) at an irradiation wavelength  $\lambda$ ),  $a$ ,  $b$ ,  $c$  and  $d$  are constants. The estimated values of the constants  $a$ ,  $b$ ,  $c$  and  $d$  are tabulated in Table 1.

Although the film dosimeters should be calibrated at the wavelength of interest, it may be useful, in case of absence of calibration facilities at wavelength of interest, to use a calibration at a certain irradiation wavelength (e.g. 298.8 nm) for determination of radiance exposure at any other irradiation wavelength. This can be accomplished by using a correction factor related to the sensitivity of the TB/PVB films towards the UV irradiation wavelength. This correction factor, which depends on irradiation wavelength, is referred to as the relative irradiation wavelength response, or relative UV spectral sensitivity,  $K(\lambda)$ , normalised to unity at the irradiation wavelength of calibration (e.g. 298.8 nm). Accordingly, the calibration curve at 298.8 nm of TB/PVB films can be written in a general form for the assessment of the UV radiance exposure at any irradiation wavelength as follows [16–20,22]:

$$K(\lambda)D = 1.915 + 36.6x - 53.7x^2 + 35.68x^3 \quad (2)$$

where  $K(\lambda)$  is the relative irradiation wavelength response of the film that normalised to unity at 298.8 nm irradiation wavelength.

The irradiation wavelength response,  $K(\lambda)$ , of the TB/PVB film containing 34 phr chloral hydrate at different irradiation wavelengths were obtained by calculating the required dose to induce change in absorbance at 551 nm (i.e.  $\Delta A_{551}$ ) equal to 0.25, 0.5 and 0.75. For example, from Fig. 2, it was found that the required dose to induce  $\Delta A_{551} = 0.5$  at 201, 248.5, 298.8, and 366 nm irradiation wavelengths is equal to 0.35, 5.7, 11.1, and  $35.3 \text{ kJ m}^{-2}$ , respectively.

Table 2

 $K(\lambda)$  values at different irradiation wavelengths for TB/PVB films ([chloral hydrate] = 34 phr)

$\lambda$ (nm)	$K(\lambda)$
201.2	$31.7 \pm 1.6$
248.5	$1.95 \pm 0.09$
298.8	$1 \pm 0.05$
366	$0.314 \pm 0.006$

The relative response,  $K(\lambda)$ , was evaluated at each irradiation wavelength by employing Eq. (2) by setting  $x$  equal to 0.25, 0.5 or 0.75 with their corresponding  $D$  values. The average values of  $K(\lambda)$  at different irradiation wavelengths are tabulated in Table 2.

The expressions given by Eq. (2) are represented by the response curves of TB/PVB films at different irradiation wavelengths in Fig. 2. The average deviation of the experimental points (i.e. UV incident energy), from the response curves given in Fig. 2 is about  $\pm 5\%$ . Therefore, Eq. (2) can be used to quantify the UV dose received by TB/PVB films for a given change in response ( $\Delta A_{551}$ ), at any irradiation wavelength. It should be mentioned that calibrations are necessary for the conditions where the films are going to be used and the previous evaluations should be used only in case of absence of calibration facilities at the wavelength of interest.

The resulting  $K(\lambda)$  values were plotted as a function of irradiation wavelength (Fig. 3). It can be seen that the sensitivity of the film increases gradually with the decrease of irradiation wavelength and reaches a maximum at 201 nm irradiation wavelength. This trend is similar to that obtained by Abdel-Fattah et al. [19] with BPB/PVB films.

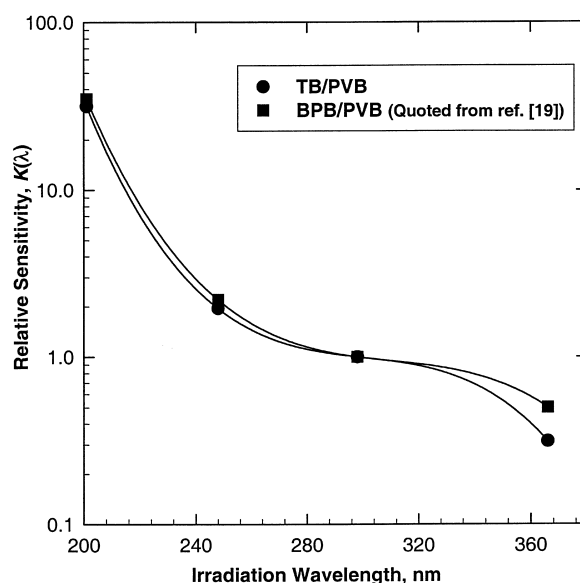


Fig. 3. Relative spectral sensitivity of TB/PVB films ([chloral hydrate] = 34 phr) and BPB/PVB films ([chloral hydrate] = 33.3 phr; quoted from [19]).

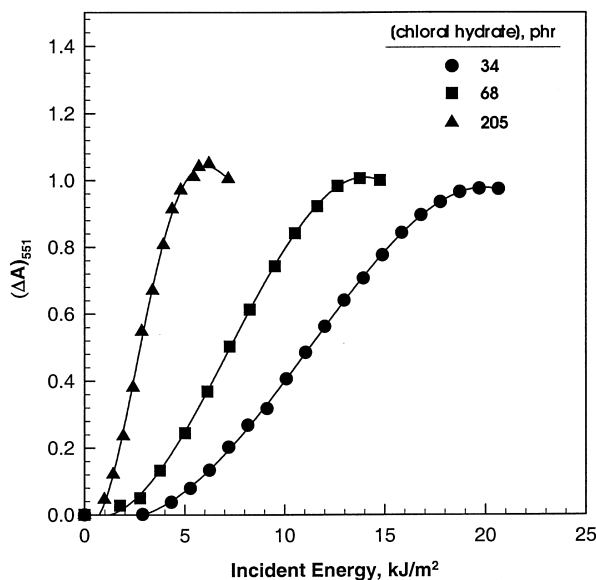


Fig. 4. Variation in  $(\Delta A)_{551}$  at 551 nm of TB/PVB films containing different concentrations of chloral hydrate with incident UV dose at an irradiation wavelength of 298.8 nm.

### 3.3. Effect of chloral hydrate concentration on dose response

The effect of a change in the concentration of chloral hydrate on the dose response of TB/PVB films was studied by using films containing 34, 68 and 205 phr of chloral hydrate. Fig. 4 shows the response curves of TB/PVB films containing different concentrations of chloral hydrate using an irradiation wavelength of 298.8 nm. Obviously, all curves show the same behaviour, namely they have S-shape and reach saturation at higher doses, but they differ in the initial response value (slope). It can be seen that, the suitable range of UV-dose depends solely on the concentration of chloral hydrate in the film.

A plot of the maximum range of response (i.e. dose at which the film changes colour completely from yellow to red) versus the concentration of chloral hydrate is shown in Fig. 5. It can be seen that, the useful range decreases exponentially with the increase of concentration of chloral hydrate. From this figure, one can predict the suitable concentration of chloral hydrate in the film for any desired range of UV-dose and also the dose at which the film will change its colour from yellow to red.

### 3.4. Assessment of uncertainty

To be meaningful, a measurement of UV incident energy shall be accompanied by an estimate of the uncertainty in the measured value. Factors contributing to the total uncertainty may be separated into two types, type A and type B. Type A is mainly related to the calibration and type B is associated mainly with the measuring equipment and the films.

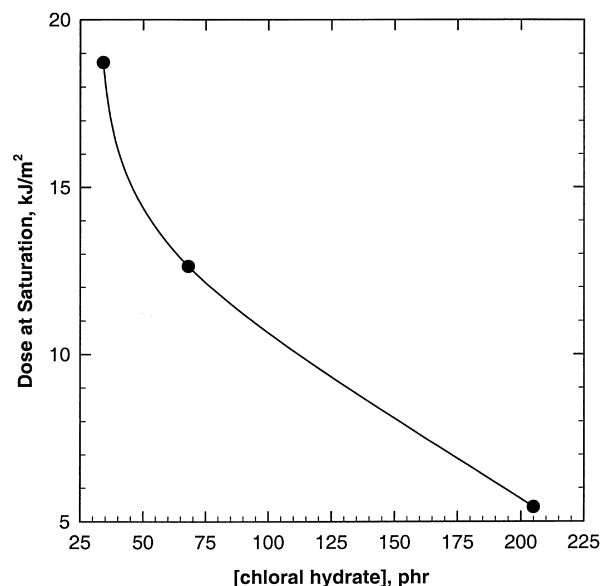


Fig. 5. Dose at saturation as a function of the concentration of chloral hydrate at an irradiation wavelength of 298.8 nm.

The reproducibility of the Unicam UV-4 spectrophotometer was determined by reading the absorbance value of an irradiated film (at 551 nm wavelength, bandwidth 2 nm and absorbance level of 0.7) several times (100 readings per film). From the data obtained, it was found that the coefficient of variation ( $1\sigma$ ) is  $\pm 0.3\%$ , reflecting the precision of the spectrophotometer. The uncertainty of the energy values as a result of the reproducibility of the irradiance meter, stopwatch and handling was found to be 1%. The error of the stability of the geometry of the UV-source is assumed to be 2%. The reproducibility of the measurement of several films irradiated to the same dose (10 times for each film) was found to be 1% ( $1\sigma$ ).

On the other hand, the type A uncertainty (at one standard deviation, i.e.  $1\sigma$ ) arising during calibration of TB/PVB film ([chloral hydrate] = 34 phr) using the irradiation wavelength 298.8 at 551 nm measurement wavelength. Four replicate measurements of absorbance were made at each value of incident energy and 19 incident energies were applied in the range 1.44–19.68  $\text{kJ m}^{-2}$ , i.e. 76 replicates were performed. It was found that, the type A uncertainty ( $1\sigma$ ) associated with the measurement of dose response at 551 nm is 2.2% [23].

The combined uncertainty ( $U_c$ ) is calculated through combining all components in quadrate at one standard deviation ( $1\sigma$ ) as follows:

$$U_c = \sqrt{(0.3)^2 + (1)^2 + (2)^2 + (1)^2 + (2.2)^2} = 3.3\%$$

The combined uncertainty (at two standard deviations, i.e.  $2\sigma$ , approximately equal to a 95% confidence level) is found by multiplication of  $U_c$  (at  $1\sigma$ ) by two. Hence, the combined uncertainty using TB/PVB films is 6.6%. In other words, the error associated with the determination of an incident

energy,  $D$ , (at a confidence level of 95%) using TB/PVB films at our experimental conditions not exceed 7%.

The standard deviation  $dD$  of the estimated exposure dose  $D$  is given by differentiating Eq. (2) as follows:

$$dD = \frac{1}{K(\lambda)} \frac{\delta D}{\delta x} dx \quad (3)$$

where  $dx$  is the standard deviation of  $x$  and equals to  $0.066x$ .

For an irradiation wavelength of 248.5 nm,  $K(\lambda)$  is 1.95 and so

$$\begin{aligned} dD &= \frac{1}{1.95} (36.6 - 107.4x + 107.04x^2) dx \\ &= \frac{0.066}{1.95} x (36.6 - 107.4x + 107.04x^2) \end{aligned} \quad (4)$$

As an example, it is assumed that a TB/PVB film containing 34 phr chloral hydrate is exposed to a dose  $D$  at an irradiation wavelength of 248.5 nm which induces a change in absorbance  $\Delta A_{551} = 0.6$ . The value of the dose  $D$  can be calculated using Eq. (2) as to be  $6.3 \text{ kJ m}^{-2}$ . The standard deviation of  $D$  ( $dD$ ) in turn can be calculated using Eq. (4) to be 0.22. Accordingly, the estimated dose inducing a change in absorbance of TB/PVB film of 0.6 at 551 nm is  $6.3 \pm 0.22$ . The standard error of dose determination in this example at a confidence level of 95% is  $\pm 3.5\%$ .

### 3.5. Post-irradiation stability

Two irradiated TB/PVB films ([chloral hydrate] = 34 phr) with an exposure dose of  $11 \text{ kJ m}^{-2}$  at 298.8 nm irradiation wavelength at room temperature were stored immediately after irradiation, one in dark and the another in indirect day light. The films were measured spectrophotometrically at 551 nm wavelength at different intervals of time during the post-irradiation storage period of 60 days. Fig. 6 shows

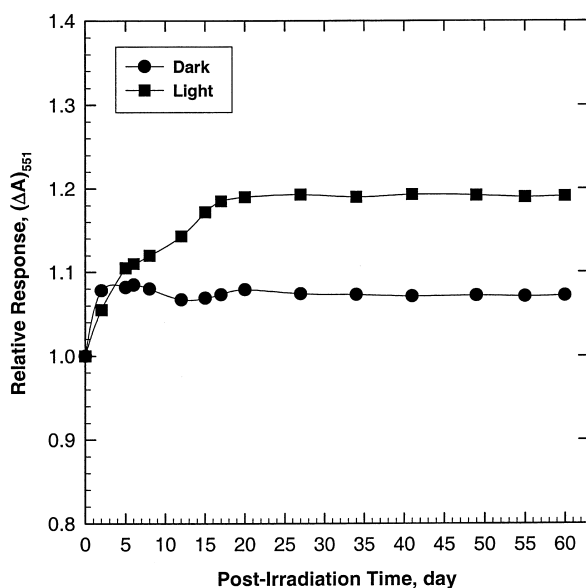


Fig. 6. Post-irradiation stability of TB/PVB films stored in the dark and indirect daylight. Exposure dose of  $11 \text{ kJ m}^{-2}$  at 298.8 nm.

change in  $\Delta A_{551}$  relative to the value at zero time as a function of the storage time. It can be seen that,  $\Delta A_{551}$  of the film stored in light increases gradually with about 20% during the first 20 days of storage and then tend to be stable to the end of the storage period. On the other hand, the film stored in dark shows an increase in  $(\Delta A)_{551}$  of about 8% during the first 24 h of storage and then tends to be stable to the end of the storage period.

## 4. Conclusions

TB/PVB film dosimeters prepared from poly(vinyl butyral) and thymol blue indicator in the presence of chloral hydrate as the radiation sensitive element have been developed and investigated for ultraviolet radiation actinometry. These films are thin (approximately 0.07 mm), strong, flexible and of good optical quality. They change their colour from yellow to red when exposed to ultraviolet radiation. The dose at which the films change colour, i.e. useful range, depends on the concentration of chloral hydrate and the irradiation wavelength. The sensitivity of the films toward UV-irradiation increases exponentially with the decrease in irradiation wavelength and reaches maximum at 201.2 nm. The percent uncertainty of estimating incident energy by the films at two standard deviations ( $2\sigma$ ) are found to be 6.6%. The film dosimeters show good post-irradiation stability when stored in dark. This dosimeter, if suitably filtered, may provide the basis for a UV badge with many medical and industrial applications in UV-A, UV-B and/or UV-C regions of the spectrum.

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